IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No.	:	10/549,670
Applicant	:	Stephen Moreton
Filed	:	July 3, 2006
Title	:	SILICA-BASED INDICATING DESICCANTS
	:	
	:	
Confirmation No.	:	3701
Group Art Unit	:	1797
Examiner	:	Bryan T. Kilpatrick
	:	
Docket No.	:	0068905-000267

BRIEF ON APPEAL

Real Party in Interest

The real party of interest is PQ Silicas UK Limited, having a place of business at Bank Quay, 4 Liverpool Road, Warrington, Cheshire, WA5 1AQ.

Related Appeals and Interferences

None.

Status of Claims

Claims 1 and 4-35 are currently pending in the application. Claims 2 and 3 were cancelled.

Pursuant to a final Office Action mailed on December 31, 2009, claims 1, 4-11, 19-29, 32, and 34-35 stand rejected under 35 U.S.C. § 102 as allegedly anticipated by International Patent Application Publication No. WO02/057772, to Moreton (the "'772 publication"). Claims

1 and 4-35 stand rejected under 35 U.S.C. § 103 as allegedly obvious over the '772 publication. Those rejections were repeated in a July 20, 2010 Advisory Action.

All pending claims are on appeal and are reproduced in the claims Appendix A.

Status of Amendments

The Office Action appealed from was issued on December 31, 2009, in which all pending claims were rejected (see grounds above). A subsequent Advisory Action (mailed March 11, 2010) stated that all amendments proposed in the March 1, 2010 Response After Final Rejection were entered, but no amendments were presented in that response. Additional amendments were presented in a response filed on June 30, 2010. The Advisory Action mailed on July 20, 2010 states that those amendments were entered.

Summary of the Claimed Subject Matter

As shown more fully below by reference to the original specification made below in the context of the claim, independent claim 1 is directed to a silica-based material desiccant using iron and bromine as an active indicator. The desiccant indicates humidity at a relative humidity below 20% by a color change. The desiccant is essentially copper-free or copper is present in an amount less than 0.002% by weight with respect to the anhydrous silica-based material.

Independent claim 23 is directed to an indicating desiccant for indicating humidity at a relative humidity below 20% by a color change. The desiccant comprises a silica-based material provided with, as the active indicator system, separate sources of iron and bromide. The desiccant is essentially copper-free, or when copper is present it is in an amount which is less than 0.002% by weight with respect to the anhydrous silica-based material. The iron source is provided by one or more salts selected from the group consisting of iron(II) sulphate, iron(III) chloride, iron(III) nitrate, iron(III) sulphate, ammonium iron(II) sulphate, ammonium iron(III) sulphate and potassium iron(III) sulphate.

Independent claim 29 is directed to a method for preparing an indicating desiccant by impregnating a silica-based material with a source of iron and a source of bromide to produce a product that is essentially copper-free and in which the iron and bromide are active indicators.

Independent Claims 1 and 29

Claims 1, 23, and 29 are the only independent claims under appeal.

1. An **indicating desiccant for indicating humidity at a relative humidity below 20% by a color change** (Specification, page 6, lines 9-12) comprising **a silica-based material** (Specification, page 3, lines 21-25) provided with, **as the active indicator system** (Specification, page 3, lines 10-11), **a source of iron** (Specification, page 3, lines 12-14 and 29-38) and **a**

source of bromide (Specification, page 4, lines 1-6), wherein **the desiccant is essentially copper-free** (Specification, page 3, line 17), or **when copper is present it is in an amount which is less than 0.002% by weight with respect to the anhydrous silica-based material** (Specification, page 3, lines 18-20).

23. An **indicating desiccant for indicating humidity at a relative humidity below 20% by a color change** (Specification, page 6, lines 9-12) comprising a **silica-based material** (Specification, page 3, lines 21-25) provided with, **as the active indicator system** (Specification, page 3, lines 10-11), **a source of iron** (Specification, page 3, lines 12-14 and 29-38) and **a source of bromide** (Specification, page 4, lines 1-6), wherein **the desiccant is essentially copper-free** (Specification, page 3, line 17), or **when copper is present it is in an amount which is less than 0.002% by weight with respect to the anhydrous silica-based material** (Specification, page 3, lines 18-20), in which **the iron source is provided by one or more salts selected from the group consisting of iron(II) sulphate, iron(III) chloride, iron(III) nitrate, iron(III) sulphate, ammonium iron(II) sulphate, ammonium iron(III) sulphate and potassium iron(III) sulphate** (Specification, page 3, lines 29-34).

29. A method of preparing **an indicating desiccant** (Specification, page 4, lines 28-29) comprising **impregnating a silica-based material** (Specification, page 5, lines 18-21) with **a source of iron** (Specification, page 4, lines 34-37) and **a source of bromide** (Specification, page 5, lines 12-17) to produce an **essentially copper-free product** (Specification, page 3, line 17) in which the **iron and bromide are the active indicators** (Specification, page 3, lines 10-11).

Grounds of Rejection to Be Reviewed on Appeal

1. Whether claims 1, 4-11, 19-29, 32, and 34-35 were properly rejected under 35 U.S.C. § 102 as anticipated by the '772 publication.

2. Whether claims 1 and 4-35 were properly rejected under 35 U.S.C. § 103 as obvious over the '772 publication.

Argument

I. Rejection under 35 USC. § 102

In an Office Action mailed December 31, 2009, claims 1, 4-11, 19-29, 32, and 34-35 were rejected under 35 U.S.C. § 102 as anticipated by International Patent Application Publication No. WO02/057772, to Moreton (the "'772 publication"). Applicant respectfully disagrees.

Claims 1, 4-11, 19-22, 24-28, and 35

A. The '772 publication does not teach the copper-related limitation of the claims.

The indicating desiccant of claim 1 and its dependent claims (with the exception of claims 34 and 35) "is essentially copper-free, or when copper is present it is in an amount which is less than 0.002% by weight with respect to the anhydrous silica-based material." Nothing in the '772 publication teaches a combination of Fe and Br salts to provide an indicator that works below 20% relative humidity with a copper level below 0.002% by weight.

The '772 publication can not anticipate the claims of the instant application, because it does not disclose the claimed copper range with sufficient specificity to constitute an anticipation of the claims. *See, e.g., Atofina v. Great Lakes Chem. Corp.*, 441 F.3d 991, 999, 78 U.S.P.Q.2d 1417, 1423 (Fed. Cir. 2006). Although the '772 publication states that "the source of copper…is up to 0.5 per cent by weight of the silica-based material," ('772 application, page 2, lines 23-25), the '772 publication states that the lowest useful range of copper is "in the range 0.002 to 0.1 per cent by weight." ('772 application, page 2, line 26). The successive ranges recited in the '772 publication only increase the amount of copper, they do not decrease it. ('772 application, page 2, lines 26-28).

In the "Response to Arguments" section of the December 31, 2009 Office Action, the Examiner asserted that the disclosure of "**up to** 0.5 percent ... expressly encompasses copper-free and less than 0.002% by weight." (Office Action, pages 10-11) (emphasis in original). That argument ignores the express teachings and clear intent of the '772 publication. The '772 publication purports to be "A new system *based on copper salts*...." ('772 publication, page 2, line 6) (emphasis added). All of the stated embodiments of the '772 publication include copper. The abstract of the '772 publication states that the indicating desiccant includes "a source of copper."

The '772 publication's disclosure of "up to 0.5%" can not teach claim limitations that are "essentially copper free" or "less than 0.002% by weight," because the "up to 0.5%" must be read in the context of the remainder of the '772 publication's disclosure, and that context explicitly carves out any embodiments without copper.

In the March 11, 2010, Advisory Action, the Examiner states that the requirements of § 102 are met by the disclosure of "up to 0.5% copper." This ignores the plain meaning of both the remaining disclosure of the '772 publication and of the *Atofina* decision.

The '772 publication does not teach a composition where copper is an optional component that may be present "up to" a given percentage, but where the composition would still function if copper were excluded entirely. If the composition of the '772 publication were copper-free, or if it had less than 0.002% copper, the entire disclosure would be frustrated. The '772 publication should not and can not be read to teach both what it teaches (that copper is required) and its opposite (that copper is excluded).

B. The '772 publication does not teach or suggest a color change below 20% relative humidity.

Claim 1 and its dependent claims require a color change below 20% relative humidity. The '772 publication, on the other hand, limits the color change to an equilibrium relative humidity "between 20 to 30 per cent." ('772 publication, page 3, lines 13-14). The reason for this is clear: the desiccant of the '772 publication is intended to reduce the relative humidity of a gas to below 30%, then the desiccant is to be replaced. ('772 publication, page 3, lines 13-15).

The "below 30%" statement in the '772 publication should not be cited to anticipate the "below 20%" limitation in the instant application, because the "below 30%" statement in the '772 publication is relevant only to reduction of relative humidity, not to indication of the reduction of relative humidity. For that purpose, the '772 publication only provides the "between 20 to 30 per cent" limitation. Moreover, the cited range and the claimed range do not overlap.

In the March 22, 2010, Advisory Action, the Examiner states that the '772 publication "discloses a desiccant that is capable of reducing relative humidity below about 30%." The Examiner concludes that "reducing relative humidity below about 30%" gives access to an open range that includes "indicating humidity at a relative humidity below 20% by a color change" because 20% is less than 30%. This is incorrect.

The "below 30%" statement in the '772 publication (page 3, lines 11-12) merely indicates how much the desiccant may reduce relative humidity. That is irrelevant to the claimed invention, which does not claim reduction of relative humidity to a specific level. Instead, the claims are directed to the level of relative humidity at which the desiccant actually shows that relative humidity has been successfully reduced. Even if the desiccant of the '772 publication were to reduce the relative humidity to a level below 30% - for example 5% (and this

hypothetical is not an admission that it does) - the '772 publication only contemplates showing that a reduction to between 20% and 30% has occurred. Presumably the '772 publication's desiccant would change color between 20% and 30%, then continue to reduce relative humidity. The claimed invention, on the other hand, changes color when the relative humidity is "below 20%." Clearly this is different from the purported teachings of the '772 publication.

Claim 23

Claim 23 is patentable at least for the reasons given in the prior section discussing claims 1, 4-11, 19-22, 2428, and 35, which is incorporated by reference herein. It is separately patentable at least because it requires that the indicating desiccant comprise a source of iron selected from a specific list of salts and, separately, it requires a source of bromide. Because the source of iron is limited to the specific list of salts, none of which include a bromide, the source of iron and the source of bromide must be different.

Iron halides are prone to degradation caused by hydrolysis upon heating. The indicating desiccant of this claim allows regeneration by heating to remove water. This is explained in the application as filed; for instance on page 6 it is explained that the color change is reversible when the desiccant is dried and the desiccant can be regenerated at least once and often many times for further use.

The '772 publication is only concerned with a silica-based material having impregnated thereon a source of copper and a source of bromide in a method for preparing an indicating desiccant. There is no indication in this citation of the use of a source of iron as the basis for an indicator system in combination with a salt which is a source of bromide. The '772 publication neither teaches nor suggests all of the limitations of claim 23.

Claims 29, 32, and 34

Claims 29, 32, and 34 require that the claimed composition or prepared product be "essentially copper free." No option is given for inclusion of even as much as less than 0.002% copper by weight. Therefore, the Office Action's already erroneous reasoning leading to the conclusion that the '772 patent teaches all of the limitations of the claims is further exacerbated by application of that reasoning to a claimed composition that always must be "essentially copper free." The requirement that the claimed product be "essentially copper free" simply can not be reconciled with an allegedly anticipating document that is based entirely on inclusion of copper. The additional arguments presented above for claims 1, 4-11, 19-22, 24-28, and 35 are incorporated by reference herein.

II. Rejection under 35 U.S.C. § 103

In an Office Action mailed December 31, 2009, claims 1 and 4-35 were rejected under 35 U.S.C. § 103 as obvious over the '772 publication. Applicant has already argued, above, that the limitations of the claims are neither taught nor suggested by the '772 publication. Those arguments are incorporated by reference into this section of the Brief as if fully repeated herein. Further, for those claims that the Office Action did not consider to be anticipated (including claims 12-18, 30, 31, and 33), the applicant hereby argues that the limitations of those claims are neither taught nor suggested by the '772 publication.

Moreover, there is no motivation to decrease the level of the indicator below 20% in the '772 publication, because the desiccant is only intended to be effective to a level below 30 per cent - that is, just below 30 per cent - and not, significantly, a level "up to" 30 per cent, which the '772 publication could have included if such were the intent.

Claims 1, 4-21, 24-28, and 35

Applicant has already argued in section I, above, that the limitations of claims 1, 4-11, 19-28, and 35 are neither taught nor suggested by the '772 publication. For at least those same reasons the rejection of those claims and of claims 12-18 are also infirm.

A. The requirement that the '772 publication be copper-based teaches away from the claimed invention.

In addition to the reasons already given, the rejection under § 103 is also untenable because the '772 publication teaches away from the claimed invention. It is well-established that the fact that a reference teaches away from a claimed invention is a significant factor in finding that a combination is no obvious. *See In re Gurley*, 27 F.3d 551, 554, 31 USPQ2d 1130, 1132 (Fed. Cir. 1994). There could not be a more clear example of "teaching away" from an invention than that provided by the '772 publication, where the '772 publication requires copper for a copper-based system, and where the claims of the invention are "essentially copper-free, or when copper is present it is in an amount which is less than 0.002% by weight."

B. One skilled in the art would not be motivated to modify the '772 publication in the ways suggested by the Examiner.

Any alleged *prima facie* case of obviousness is also negated by the fact that there is no motivation to decrease the level at which the color of the desiccant changes below 20% in the '772 publication. As already noted, the desiccant of the '772 publication is only intended to be effective to a level below 30 per cent - that is, just below 30 per cent - and not, significantly, a level "up to" 30 per cent, which the '772 publication could have included if such were the intent. There is no evidence that one skilled in the art would have been motivated to use that disclosure

to engineer a compound with capabilities so far removed from those that are reported in the '772 publication.

C. Reduction of copper in the '772 publication would not lead to a reduction in the ability to work below 20% relative humidity.

Even if the '772 publication could be read to teach a desiccant that "is essentially copper-free, or when copper is present it is in an amount which is less than 0.002% by weight," or if it could be read to teach "indicating humidity at a relative humidity below 20% by a color change" (both of which applicant denies), it could not be read to teach both at once. The '772 publication teaches that a higher Br:Cu ratio leads to an increase in the relative humidity at which a color change occurs. ('772 publication, page 13, lines 10-11).

Even if one skilled in the art were motivated by the '772 publication to prepare a desiccant with a color change that occurs below 20% relative humidity, he would not do so by decreasing or eliminating copper. Instead, he would increase the amount of copper, decreasing the Br:Cu ratio and decreasing the percentage of relative humidity at which the color change occurs. This further demonstrates that the '772 publication teaches away from the claimed invention.

D. Iron provides the active indicator system in the instant application, and use of iron leads to unexpected results.

One of skill in the art would have no expectation of success in modifying the '772 publication to arrive at the claimed invention. In the '722 publication, the active indicator system is a copper-based system. The claimed invention, on the other hand, has an active indicator system provided by the iron and bromide. An iron-bromide system is neither taught nor suggested by the '772 publication.

In the Advisory Action mailed on March 11, 2010, the Examiner stated that "the desiccant [of the '772 publication] can employ the use of dyes or other coloring materials (p. 3 [of the '772 publication] that can comprise iron salts (p. 3)…)." The Examiner has provided no reason why one of skill in the art would conclude that a compound providing an aesthetic effect should also serve as the basis of an indicator system. The '772 publication clearly considers iron to be only another way that the indicator may be colored. "Addition of blue dyes can impart a blue colour to the humidified substrate and addition of iron (III) salts, can impart a yellow colour to the humidified substrate." ('772 publication, page 3, lines 33-34).

A showing of unexpected results rebuts a *prima facie* case of obviousness. Selection and use of iron as the active indicator system leads to unexpected results. *See In re Waymouth*, 499 F.2d 1273, 1276, 182 USPQ 290, 293 (CCPA 1974). This is demonstrated in the attached Declaration of Stephen Moreton, Ph.D. (the inventor). That Declaration is provided in Appendix B of this Brief and was first submitted in a Response filed on September 1, 2009. The Declaration was entered into the record by the Examiner on page 2 of the December 31, 2009 Office Action. The Declaration demonstrations that the claimed combination of iron salts and bromine allows accurate indication of humidity below 20% relative humidity. This is clearly shown in the comparison of the controls with the inventive compositions in Tables 1-6 and Exhibits 1-6 of the Declaration.

Claim 22

Claim 22 is patentable over the '772 publication as applied under 35 U.S.C. § 103 at least for the reasons given above for patentability of claims 1, 4-21, 24-28, and 35. Those arguments are incorporated by reference herein. Claim 22 is also separately patentable due to the unexpected results noted by Dr. Moreton in his declaration, which is provided as Appendix B

and which was originally submitted for consideration on September 1, 2009. Claim 22 claims "A desiccant as claimed in claim 1 in which the source of iron is an iron (III) salt or salts." Tables 1, 3, 4, and 5 of the Declaration provide particularly relevant information. All of those Tables include results from test that used iron (III) salts, and all three of them provide exceptional color changes below RH 20%.

This color change is unexpected, described by Dr. Moreton as a "major improvement" or "marked increase" in the color change provided by the iron (III) desiccant when combined with a source of bromine at low relative humidity when compared with a control that does not include bromine. This evidence rebuts any prima facie case of obviousness (assuming one exists, which Applicant does not admit) over the '772 publication, and provides a separate basis for patentability of claim 22.

Claim 23

For the reasons given in the discussion of the rejections under 35 U.S.C. § 102, the '772 publication does not teach or suggest all of the limitations of claim 23. The rejection under 35 U.S.C. § 103 must therefore also fail. The above arguments related to the inapplicability of a 35 U.S.C. § 103 rejection to claims 1, 4-21, 24-28, and 35 are also incorporated by reference here. Moreover, as set out in the introduction to the present application, an aim of the application is to provide indicators which function at low relative humidity in a desiccant of silica and which are not toxic. Starting from copper salts as the indicator system, such as set out in the '772 publication, would not be considered as a sensible starting point by one skilled in the art. There would be no reason to refer to or incorporate the teachings of the '772 publication.

Nothing in the '772 publication teaches or suggests the beneficial effect of using a source of bromide separate from the source of iron. The chemical environment within a silica gel will

have a considerable effect upon the indicating behavior of salts incorporated within it. A salt which is taught as a background color dye for inclusion in included in one indicator system is *not* taught as having the ability to act as an indicator system which has suitable low relative humidity-indicating behavior in the chemical environment of a silica gel. Predicting the behavior of indicating salts within a silica gel is by no means straightforward. One skilled in the art would not have found anything in the '772 publication that teaches or suggests the beneficial effect that addition of a source of bromide to an iron system would have in creating a desirable composition.

Absent reference to the instant application, one skilled in the art who was attempting to identify an indicator system which would function in the chemical environment of a silica gel and which provides a desiccant which can be regenerated by heating would have no reason to combine a source of iron (acting as the primary indicator) modified by the inclusion of a soluble source of bromide. Claim 23 reflects the discovery by the inventors that a soluble salt acting as a source of bromide can be used to modify the relative humidity range at which iron indicators function, so that they can be used as an indicator system in a regenerated silica gel desiccant. Claim 23 is separately patentable.

Claims 29-34

Applicant has already argued, above, that the limitations of claims 29, 32, and 34 are neither taught nor suggested by the '772 publication. Those arguments are incorporated by reference herein.

All of the claims in this group require that the claimed composition or prepared product be "essentially copper free." No option is given for inclusion of even as much as less than 0.002% copper by weight. The arguments given above for claims 1, 4-28, and 35 also apply to

these claims, only moreso, because "essentially copper free" is quite clearly not contemplated by a copper-based system, and no modifications of a copper-based system could lead to a result that is otherwise. The remaining arguments in favor of patentability of claims 1, 4-28, and 35 are also incorporated by reference herein.

III. Conclusion

The rejection of the claims under 35 USC. §§ 102 & 103 should be reversed for any one of many reasons. The '772 publication does not teach or suggest an essentially copper-free desiccant or a desiccant that includes less than 0.002% by weight. The '772 publication does not suggest use of an iron-based system, which the instant invention applies to yield unexpected results; instead, the '772 publication's use of iron is limited to aesthetic purposes. The '772 publication does not teach separate sources of iron and bromide. Even if the '772 publication were otherwise relevant, one skilled in the art would only read it to suggest an increase in the amount of copper would lead to a decrease in the resulting relative humidity. Finally, nothing in the '772 publication teaches that a color change should occur at relative humidity below 20%, or even that such a thing would be desirable.

It is requested that a speedy allowance of this application be granted.

Therefore, the Applicants request a reversal of the Examiner's rejection of all pending claims, namely 1 and 4-35, under 35 USC. § 103(a), and of claims 1, 4-11, 19-29, 32, and 34-35 under 35 U.S.C. § 102.

Respectfully submitted,

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Dated: July 28, 2010

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Claims Appendix A

1. An indicating desiccant for indicating humidity at a relative humidity below 20% by a color change comprising a silica-based material provided with, as the active indicator system, a source of iron and a source of bromide, wherein the desiccant is essentially copper-free, or when copper is present it is in an amount which is less than 0.002% by weight with respect to the anhydrous silica-based material.

2-3. (Cancelled)

4. A desiccant as claimed in claim 1 in which the source of iron is present in an amount up to 2.0% by weight, calculated as Fe with respect to weight of the anhydrous silica-based material.

5. A desiccant as claimed in claim 1 in which the source of iron is present in an amount of up to 1.0% by weight, calculated as Fe with respect to weight of the anhydrous silica-based material.

6. A desiccant as claimed in claim 1 in which the source of iron is present in an amount of up to 0.6% by weight, calculated as Fe with respect to weight of the anhydrous silica-based material.

7. A desiccant as claimed in claim 1 in which the source of iron is present in an amount

of up to 0.45% by weight, calculated as Fe with respect to weight of the anhydrous silica-based material.

8. A desiccant as claimed in claim 1 in which the source of iron is present in an amount of at least 0.01% by weight, calculated as Fe with respect to weight of the anhydrous silica-based material.

9. A desiccant as claimed in claim 1 in which the source of iron is present in an amount of at least 0.02% by weight, calculated as Fe with respect to weight of the anhydrous silica-based material.

10. A desiccant as claimed in claim 1 in which the source of iron is present in an amount of 0.02 to 1.0% by weight, calculated as Fe with respect to weight of the anhydrous silica-based material.

11. A desiccant as claimed in claim 1 in which the source of iron is present in an amount of 0.05 to 0.3% by weight, calculated as Fe with respect to weight of the anhydrous silica-based material.

12. A desiccant as claimed in claim 1 in which the bromine content is equal to, or greater than, the amount of iron.

13. A desiccant as claimed in claim 1 in which the source of bromide is present in an amount such that the weight ratio of Br to Fe is at least 0.1:1.

14. A desiccant as claimed in claim 1 in which the source of bromide is present in an amount such that the weight ratio of Br to Fe is at least 0.5:1.

15. A desiccant as claimed in claim 1 in which the source of bromide is present in an amount such that the weight ratio of Br to Fe is at least 1:1.

16. A desiccant as claimed in claim 1 in which the source of bromide is present in an amount such that the weight ratio of Br to Fe is up to 100:1.

17. A desiccant as claimed in claim 1 in which the source of bromide is present in an amount such that the weight ratio of Br to Fe is up to 50:1.

18. A desiccant as claimed in claim 1 in which the source of bromide is present in an amount such that the weight ratio of Br to Fe is up to 20:1.

19. A desiccant as claimed in claim 1 in which the bromide source comprises a water soluble salt.

20. A desiccant as claimed in claim 1 in which the bromide source is selected from one

or more of the group consisting of alkali metal bromides, alkaline earth metal bromides, transition metal bromides and ammonium bromide.

21. A desiccant as claimed in claim 1 in which the bromide source is selected from one or more of the group consisting of sodium bromide, potassium bromide, calcium bromide, magnesium bromide, zinc bromide and ammonium bromide.

22. A desiccant as claimed in claim 1 in which the source of iron is an iron (III) salt or salts.

23. An indicating desiccant for indicating humidity at a relative humidity below 20% by a color change comprising a silica-based material provided with, as the active indicator system, a source of iron and a source of bromide, wherein the desiccant is essentially copper-free, or when copper is present it is in an amount which is less than 0.002% by weight with respect to the anhydrous silica-based material, in which the iron source is provided by one or more salts selected from the group consisting of iron(II) sulphate, iron(III) chloride, iron(III) nitrate, iron(III) sulphate, ammonium iron(II) sulphate, ammonium iron(III) sulphate and potassium iron(III) sulphate.

24. A desiccant as claimed in claim 1 in which the silica-based material is silica gel.

25. A desiccant as claimed in claim 24 in which the silica gel is a beaded silica gel.

26. A desiccant as claimed in claim 24 in which the silica gel is a granular silica gel.

27. A desiccant as claimed in claim 24 in which the silica gel is a dry or humidified gel.

28. A desiccant as claimed in claim 24 in which the silica gel has a pore volume to nitrogen in the range 0.2 to 2.0 cm^3g^{-1} and a BET surface area in the range 200 to 1500 m^2g^{-1}.

29. A method of preparing an indicating desiccant comprising impregnating a silica-based material with a source of iron and a source of bromide to produce an essentially copper-free product in which the iron and bromide are the active indicators.

30. A method as claimed in claim 29 in which the source of iron is present in an amount up to 2.0 per cent by weight, calculated as Fe with respect to weight of the anhydrous silica-based material, and the source of bromide in an amount such that the weight ratio of Br to Fe is at least 0.1:1.

31. A method as claimed in claim 29 in which a humidified silica gel containing from 20 to 30% by weight water is soaked in a solution containing from 0.1% to the saturation point of an iron salt and a source of bromide, excess solution is drained from the treated silica gel and the silica gel is dried at a temperature in the range 80ºC to 230ºC.

32. A method as claimed in claim 31 in which the gel is soaked in said solution for a period in the range of 2 to 24 hours.

33. A method as claimed in claim 30 in which impregnation is effected by mixing a humidified silica gel containing from 15 to 30 per cent moisture by weight with a solution containing a source of iron and a source of bromide, the amount of solution used being just sufficient to produce the required loading of iron and bromide on the silica gel, and subsequently drying the treated silica gel at a temperature in the range 80°C to 230 °C.

34. A desiccant as claimed in claim 1, wherein the desiccant is essentially copper-free.

35. The desiccant as claimed in claim 1, wherein copper is present in an amount less than 0.002% by weight with respect to the anhydrous silica-based material.

Evidence Appendix B

The Declaration of Dr. Steven Moreton, originally filed on September 1, 2009, and entered by the Examiner on December 31, 2009, is included herewith.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:	:	**PATENT APPLICATION**
	:	
Stephen Moreton	:	Silica-Based Indicating Desiccants
	:	
Serial No.: 10/549,670	:	Group Art Unit: 1797
	:	
Filed: July 3, 2006	:	Examiner: B. Kilpatrick

DECLARATION OF STEPHEN MORETON, Ph.D.
UNDER 37 C.F.R. § 1.132

1. I, Dr. Stephen Moreton, am the inventor of the "Silica-Based Indicating Desiccants" that are the subject of U.S. Patent Application No. 10/549,670 (the " '670 application"). In 1989 I received my Ph.D. in inorganic chemistry from Edinburgh University.

2. I have read and am familiar with the '670 application.

3. I have generated data to demonstrate the effectiveness of common iron salts in conjunction with a source of bromide, as humidity indicators at RH from 0 to 20% in silica gel desiccant.

4. Silica gels were prepared containing different iron salts together with sodium bromide using the method of example 4 in the '670 application as filed. These impregnated gels were dried for around 4 hours and exposed to air streams at 10, 20, 40 & 80 % relative humidity for 7 hours, as described in the patent. The colours of the gel were noted, and measured using a Chromameter, according to the patent method. The samples were also photographed and the photographs are appended to this Declaration as Exhibits 1-6. Details, and the results, for various iron salts are described below.

5. The humidified silica gel (2.5 – 6.0 mm diameter granules) contained approximately 23 % water (as determined by weight loss at 145 °C). For all of the examples below the

quantities of gel, and of iron and bromide salts, were calculated to give 0.24 % Fe and 0.44 % Br in the dried product, with a Br:Fe weight ratio of 1.83. This works out at 0.57 g NaBr in each case (when Br is present), and about 126 – 128 grams of humidified gel. For the nitrate, the level of bromide was doubled to give Br:Fe of 3.7 for reasons explained below.

6. Potassium iron(III) sulphate, $KFe(SO_4)_2$

This is the potassium analogue of the ammonium iron(III) sulphate, or iron alum, employed in the patent application. A solution was prepared containing 1.4275 g of this in 10 ml water. This served as the control. Another solution contained the same plus 0.5665 g sodium bromide.

7. The solutions were mixed with 128.0 & 127.3 grams of humidified silica gel respectively. After drying at 145 °C the gels were analysed, and tested as described above. Results are shown in Table 1.

Table 1. Indicating behaviour of the dried potassium iron alum gel.

Composition	% R.H.	L*	a*	b*	Colour
No Br (control)	0	46.69	+5.22	+26.51	Light orange
	10	48.54	+3.32	+30.00	Light orange
	20	50.02	+2.30	+24.98	Light orange
	40	53.63	+2.34	+16.38	Pale yellowish orange
	80	58.97	-0.39	+9.15	Pale yellowish
With Br	0	36.77	+14.15	+34.20	Amber/brown
	10	47.63	+3.74	+30.96	Light orange
	20	51.01	+2.62	+27.60	Light orange
	40	51.25	+2.39	+19.98	Pale yellowish orange
	80	59.27	-0.44	+10.54	Pale yellowish

8. It is clear from the data in Table 1 and the photographs in Exhibit 1 that the presence of the Br source gives a major improvement in colour change for the indicating desiccant between 0 and 20% RH compared to the control sample without a source of Br.

9. Ammonium iron(II) sulphate, $(NH_4)_2Fe(SO_4)_2.6H_2O$

This compound, also known as ferrous ammonium sulphate, or Mohr's salt, will react with atmospheric oxygen and oxidise to the iron(III) state during the drying stage, and so behave similarly to ammonium iron(III) sulphate, or iron alum. A solution of 1.6854 g of this in 10 ml of water was prepared (the control) and another containing the same plus 0.5665 g sodium bromide. The solutions were mixed with humidified gel and dried at 145°C and tested as above. Results are shown in Table 2.

Table 2. Properties of the dried Mohr's salt gel.

Composition	% R.H.	L*	a*	b*	Colour
No Br (control)	0	42.94	+10.70	+26.65	Light orange
	10	46.02	+10.42	+28.34	Light orange
	20	42.41	+12.28	+28.33	Light orange
	40	52.68	+3.22	+15.59	Pale orange
	80	57.47	+1.20	+14.26	Pale yellowish
With Br	0	40.18	+7.55	+31.77	Yellow/light brown
	10	40.94	+9.83	+24.44	Light amber
	20	44.40	+10.53	+28.28	Light orange
	40	51.88	+6.47	+24.99	Pale orange
	80	54.75	+1.26	+13.02	Pale yellowish

10. Again, the data in the table above, and the photographic results in Exhibit 2, show that the presence of a source of Br along with the iron(II) double salt gives a significant improvement in colour change between 0 and 20% RH.

11. Iron(III) sulphate, $Fe_2(SO_4)_2.5H_2O$ (Ferric sulphate)

A solution of 1.0530 g of this in 10 ml of water was prepared (the control) and another containing the same plus 0.5665 g sodium bromide. Each solution was mixed with humidified gel, dried at 145°C and tested as above. Results are shown in Table 3.

Table 3. Properties of the dried ferric sulphate gel.

Composition	% R.H.	L*	a*	b*	Colour
No Br (control)	0	46.49	+3.68	+28.84	Light orange
	10	49.62	+0.72	+24.38	Light yellow
	20	53.90	+0.72	+20.08	Light yellow
	40	56.12	-0.02	+13.30	Pale yellow
	80	57.28	-1.15	+6.48	Almost colourless
With Br	0	31.32	+18.26	+26.76	Deep amber/brown
	10	45.69	+5.19	+32.57	Light orange
	20	49.77	+3.54	+28.18	Light yellow
	40	54.55	+1.49	+20.71	Light yellow
	80	59.21	-1.69	+9.90	Pale yellow

12. The data in the table, and the photographic results shown in Exhibit 3, each show a marked increase in the colour change for the indicating desiccant between 0 and 20% RH.

13. Iron(III) chloride, $FeCl_3.6H_2O$ (Ferric chloride)

A solution of 1.1617 g of this in 10 ml of water was prepared (the control) and another containing the same plus 0.5665 g sodium bromide. Each solution was mixed with humidified gel and dried at 100°C for 3 hours, before being tested as above. The gentler drying condition was selected to reduce the tendency of the chloride to undergo hydrolytic decomposition.

Table 4. Properties of the dried ferric chloride gel.

Composition	% R.H.	L*	a*	b*	Colour
No Br (control)	0	49.20	-1.22	+26.98	Yellow
	10	49.77	-1.44	+31.13	Yellow
	20	53.64	-2.23	+26.54	Yellow
	40	58.91	-3.15	+15.92	Pale yellow
	80	59.49	-2.79	+8.69	Pale yellow
With Br	0	34.73	+14.92	+33.48	Deep amber/brown
	10	38.30	+10.70	+35.12	Amber
	20	45.71	+6.31	+33.79	Light orange
	40	54.85	-1.62	+24.90	Light yellow
	80	58.13	-3.28	+22.38	Light yellow

14. The data in the table, and the photographic results shown in Exhibit 4, each show a marked increase in the colour change for the indicating desiccant between 0 and 20% RH.

15. Iron(III) nitrate, $Fe(NO_3)_3.9H_2O$

Ferric nitrate was used with a lower drying temperature (80°C) and twice as much bromide than for the other salts. A solution of 1.7366 g of this in 10 ml of water was prepared (the control) and another containing the same plus 1.1330 g sodium bromide (i.e. Br:Fe weight ratio 3.7). Each solution was mixed with humidified gel and dried at 80 °C for 3 hours, before being tested as above. The gentler drying conditions, and higher bromide level, were selected to reduce the tendency of the nitrate to undergo hydrolytic decomposition. Results are shown in Table 5.

Table 5. Properties of the ferric nitrate gel dried at 80 °C.

Composition	% R.H.	L*	a*	b*	Colour
No Br (control)	0	53.49	+0.36	+17.41	Light yellow
	10	50.83	+0.68	+18.70	Light yellow
	20	55.92	-0.25	+12.92	Pale yellow
	40	59.72	-0.39	+9.26	Pale yellow, almost colourless
	80	59.94	-0.39	+6.16	Almost colourless
With Br	0	45.36	+6.71	+33.11	Amber
	10	42.86	+9.15	+35.34	Amber
	20	48.25	+3.83	+28.40	Light orange
	40	54.32	+1.44	+22.62	Light yellow
	80	55.96	-0.42	+14.61	Pale yellow

16. The data in the table, and the photographic results shown in Exhibit 5, both show a marked increase in the colour change for the indicating desiccant between 0 and 20% RH, compared to the colour change for the desiccant without the Br source.

17. Iron(II) sulphate, $FeSO_4.7H_2O$ (Ferrous sulphate)

A solution of 1.1946 g of this in 10 ml of water was prepared (the control) and another containing the same plus 0.5665 g sodium bromide. Each solution was mixed with humidified gel and dried and tested as above. Results are shown in Table 6.

Table 6. Properties of the dried iron(II) sulphate gel.

Composition	% R.H.	L*	a*	b*	Colour
No Br (control)	0	41.92	+7.76	+24.72	Amber
	10	46.54	+8.06	+28.46	Orange
	20	49.25	+6.58	+24.34	Orange
	40	52.71	+3.85	+20.75	Light orange & colourless, uneven
	80	57.96	+1.48	+14.96	Pale yellow & colourless, uneven
With Br	0	34.90	+10.18	+31.31	Deep amber
	10	45.15	+10.58	+32.81	Amber
	20	47.85	+8.55	+30.74	Orange
	40	52.05	+3.72	+19.93	Light orange & colourless, uneven
	80	58.70	+0.46	+12.87	Pale yellow & colourless, uneven

18. The data in the table, and the photographic results shown in Exhibit 6, each show a marked increase in the colour change for the indicating desiccant between 0 and 20% RH, compared to the colour change for the desiccant without the Br source.

I declare that the foregoing is true and correct, that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: September 1, 2009

S. Moreton

Stephen Moreton, Ph.D.

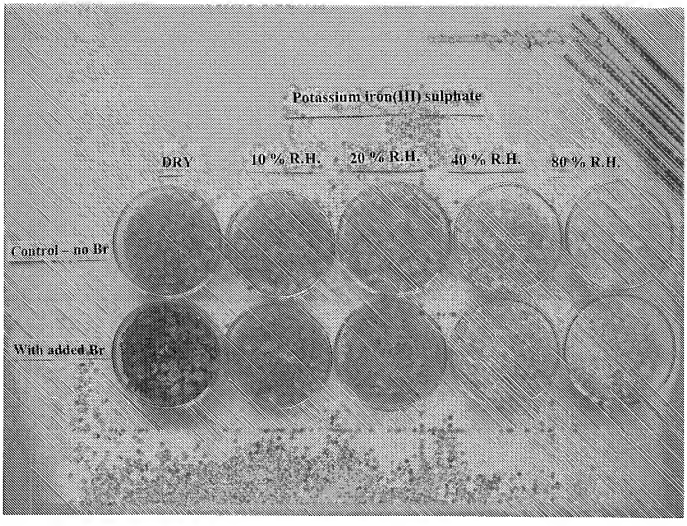
Potassium iron(III) sulphate
DRY
10 % R.H.
20 % R.H.
40 % R.H.
80 % R.H.
Control – no Br
With added Br

Exhibit 1

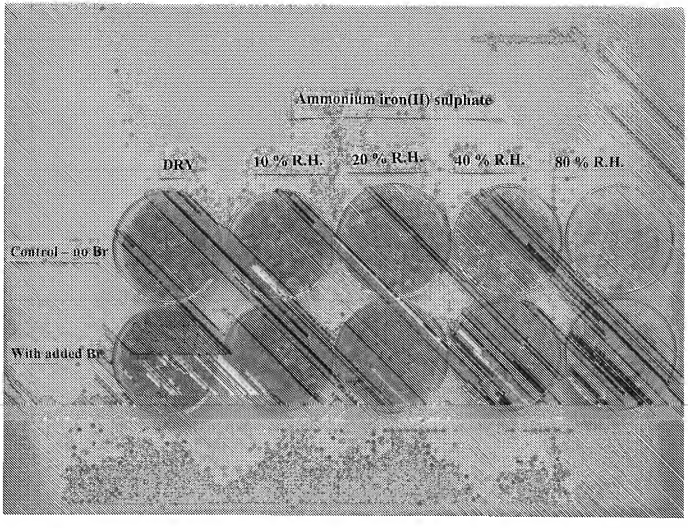
Ammonium iron(II) sulphate
DRY
10 % R.H.
20 % R.H.
40 % R.H.
80 % R.H.
Control – no Br
With added Br

Exhibit 2

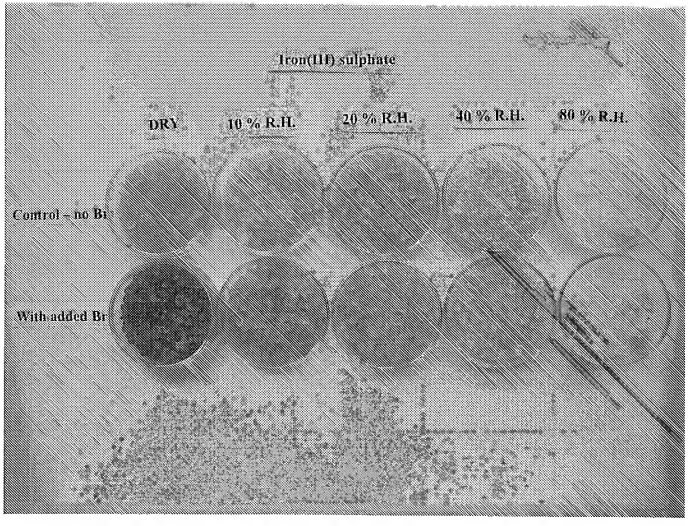
Iron(III) sulphate
DRY
10 % R.H.
20 % R.H.
40 % R.H.
80 % R.H.
Control – no Bi
With added Bi

Exhibit 3

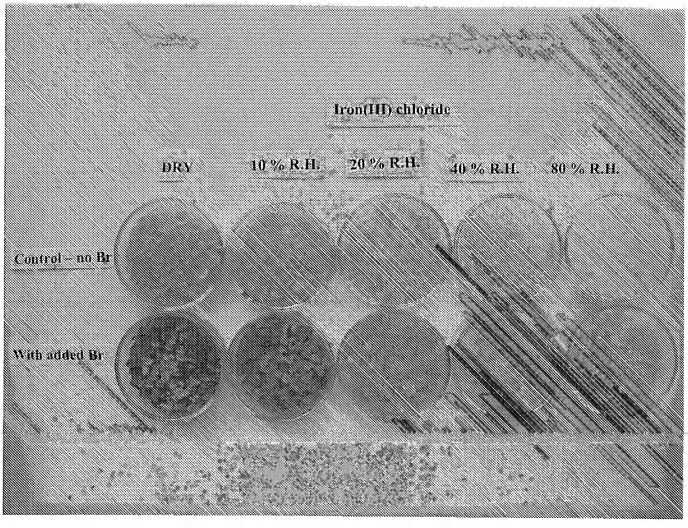
Iron(III) chloride
DRY
10 % R.H.
20 % R.H.
40 % R.H.
80 % R.H.
Control – no Br
With added Br

Exhibit 4

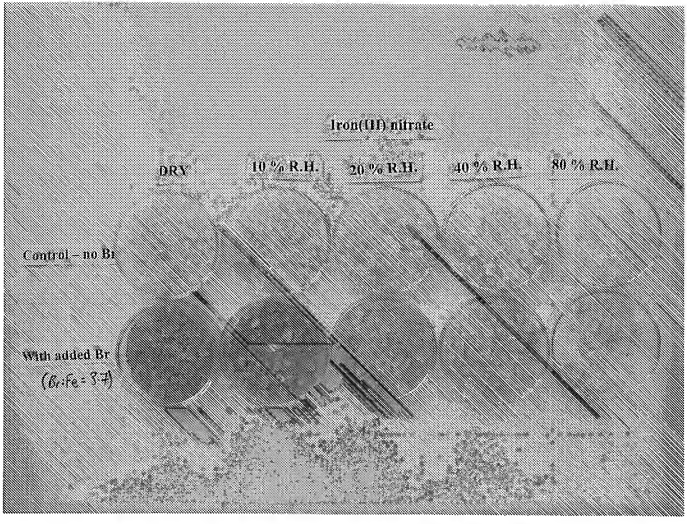
Iron(II) nitrate
DRY
10 % R.H.
20 % R.H.
40 % R.H.
80 % R.H.
Control - no Br
With added Br
(Br:Fe = 3:7)

Exhibit 5

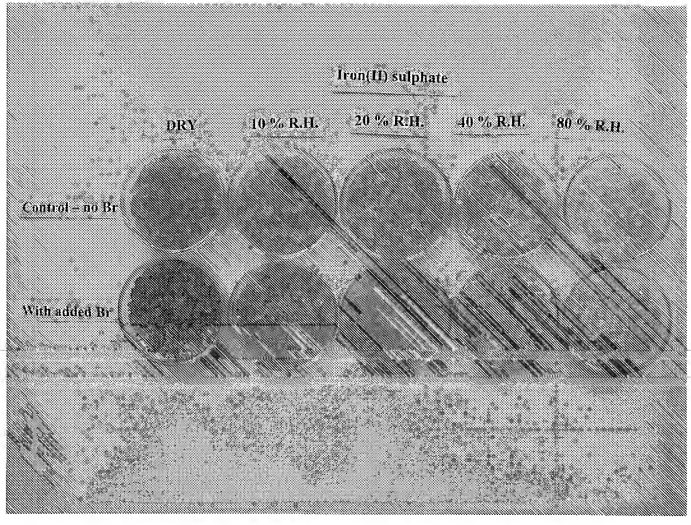
Iron(II) sulphate
DRY
10 % R.H.
20 % R.H.
40 % R.H.
80 % R.H.
Control – no Br
With added Br

Exhibit 6

Related Proceedings Appendix C

There are no present related proceedings.